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GENERATION OF LONG TIME CREEP DATA ON REFRACTORY ALLOYS AT ELEVATED TEMPERATURES

THIRD QUARTERLY REPORT

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By

J. C. Sawyer and E. B. Evans

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

LEWIS RESEARCH CENTER

UNDER CONTRACT NAS 3-2545

TRW ELECTROMECHANICAL DIVISION

THOMPSON RAMO WOOLDRIDGE INC.

CLEVELAND, OHIO

CR 54048

Third Quarterly Report
for
December 26, 1963 to March 26, 1964

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By
J. C. Sawyer and E. B. Evans
Materials Research & Development

Prepared For
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Technical Management
Paul E. Moorhead
NASA - Lewis Research Center

April 20, 1964

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INTRODUCTION

This is the third quarterly progress report on the generation of creep data of refractory metal alloys. These alloys are candidate materials for turbine and tubing applications for space electric power systems. The objectives of the program are to construct fourteen (14) creep units capable of operation at better than 1 X 10^{-8} Torr and to conduct long time creep tests over the range from 2000°F to 3200°F.

In this reporting period, the major work consisted of (a) acceptance testing of the first vacuum creep unit, (b) further bench testing of the residual gas analyzer, optical pyrometer, and optical extensometer, and (c) selection of alloys. The progress made in these areas is summarized below.

VACUUM SYSTEM

It will be recalled that the major parts of a vacuum creep unit are an upper bell jar chamber, an ion pump chamber (also functioning as the housing for internal weights) attached below the upper chamber, a bellows arrangement for external loading, and a coldwall furnace.

The first vacuum unit has been received and is now being installed. This unit, Figure 1, has been tested for 50 hours at 3500°F. Power requirements were 6 KW. A record of the pressure during the test is shown in Figure 2. The difficulties experienced during the test include temporary loss of power due to the removal of an electrical plug, heating of the sight port due to sticking shutter, and heating of coldwall due to low water pressure. All of these difficulties were corrected and the test continued.

Prior to high temperature testing, the system had been baked at 400°F and cooled with all equipment in place including weights and specimen. A base pressure of 5 X 10^{-10}mm Hg cold was measured. Accurate measurements of temperature gradients in the hot zone have not been attempted to date.

The associated equipment is shown in Figures 3-6. Figure 3 is the roughing cart, Figure 4 the hoist and bakeout cart, Figure 5 the bakeout oven, and Figure 6 the temporary console containing the temperature controller, cold junction oven, temperature recorder, and pressure gauge. A more detailed discussion of the vacuum system will be made at a later date.

RESIDUAL GAS ANALYZER

The residual gas analyzer was designed and built to monitor the residual gas composition in the vacuum creep unit during the creep run. The analyzer consists of an analyzer head attached to the vacuum creep chamber with a single portable electronic control and readout panel to service the analyzer heads on a one-at-a-time basis. At the present time only six of the fourteen chambers will be equipped with analyzer heads although attachment flanges will be provided on the other eight chambers. The readout panel not only provides the necessary actuating signals, but contains the readout equipment including an oscilloscope and recorder. An auxiliary filament power supply is provided for all six analyzer heads so that one of the two filaments available in each head can be held at a reduced temperature when not in use. By this means the life of the filament is extended.

Residual gas analyzers are made by a number of manufacturers to various designs, but all incorporate the same basic features as follows:

1. An electron source.
2. A means for accelerating the electrons which ultimately cause ionization of the residual gas.
3. A positive ion accelerator.
4. A system for discriminating between the masses of the various positive ions.
5. A positive ion detector.
6. A readout system.

1. Electron Source

All systems use a hot filament as the electron source although the design and construction of the filament varies between manufacturers. The equipment used in this case is provided with two filaments; one of thoriated tridium, which is the main filament, and a reserve filament of tungsten. The switching from one filament to another is manual. The power for the filament is supplied by either the portable electronic console or by a standby supply permanently mounted in the central console serving all vacuum units. These two power supplies operate in parallel so that when the portable console is disconnected from the analyzer head the filament in each is maintained at a reduced temperature by the standby power supply. When the portable console is connected to an analyzer head, additional power is supplied to the filament to bring it to the desired temperature. By this technique thermal shock to the filament is minimized and life of the filament is extended. Should the main filament fail then the reserve filament can be used in a similar manner. A window is provided to observe the hot filament.

2. Electron Accelerator

Electrons from the hot filament are accelerated by an electrostatic field through an ionization region defined by grids. The ionization current is 250 microamps. Unlike many of the other systems which provide a continuous stream of ions, this instrument produces ions in bunches at the rate of 10K per sec. The duration of the ionization pulse is 1.75 microseconds. This is achieved by modulating the electron beam defining grids with a pulse derived from a General Radio pulse generator and pulse amplifier. Thus, the positive ions, formed from the residual gas, are produced for only 1.75 microseconds. This is repeated every 100 microseconds.

3. Positive Ion Accelerator

The positive ions produced every 100 microseconds are removed from the ionization region by means of an electrostatic field. This principle is applied to all residual gas analyzers.

4. Ion Discriminating System

The major differences between residual gas analyzers are in the ion discriminating system. Most systems depend upon angular deflection of the ions in a magnetic field to discriminate between the ions of various masses. In the Nuclide instrument selected, the time-of-flight principle is employed. In the time-of-flight system the ions ejected from the ionization area are allowed to traverse a path of approximately 32 cm during which time the ions separate into groups dependent upon the ion masses. Thus the hydrogen ions, ejected with a higher velocity than the heavier argon ion, reach the detector sooner than the argon ions. Since the ions are produced in a finite volume, grid design and applied voltages were optimized to cause ions of like mass to bunch together during their passage down the flight tube. The ions formed farther from the detector are given a slightly greater velocity so that they will reach the detector at approximately the same time as those ions formed slightly closer to the detector. Molecules having a mass 2 through 44 (H_2 thru CO_2) can be resolved with this instrument.

5. Ion Detector

All systems measure the number of ions reaching the detector by monitoring the current flow at the detector. Since at low pressures this current is extremely small, the more sophisticated systems employ electron multipliers. The system which is used in this application embodies a 16-stage multiplier operating from a 4.3 KV power supply to boost the ion current which is fed to the readout system.

6. Readout System

The output from the electron multiplier is fed into a preamplifier operated at maximum gain. This in turn is fed into the vertical amplifier of a Hewlett Packard oscilloscope fitted with a vertical amplifier. The scanning speed of the oscilloscope is adjusted to be less than 100 microseconds and the scan is triggered by an impulse from the pulse generator previously described. Thus the horizontal beam of the oscilloscope begins to sweep at the start of the ionization pulse. This pulse can be seen on the oscilloscope by virtue of the transients associated with the start of the pulse. The end of the pulse 1.75 microseconds later can also be observed because of the associated transients. As the various positive ions arrive at the detector, they cause a vertical displacement of the beam, the magnitude of which is related to the number of ions arriving at the detector. To supplement this method of readout, the oscilloscope is equipped with a Hewlett Packard display scanner which reproduces the scope trace on a Moseley strip chart recorder. With this system residual gases at partial pressures of 10^{-12} mm Hg can be detected. All gases having masses of 2 through 44 (hydrogen--carbon dioxide) can be detected. A schematic diagram of the analyzer head is shown in Figure 7 and a block diagram of the complete system is shown in Figure 8.

The photograph, Figure 9, shows the flight tube attached to the vacuum unit. The ion source is located at the bottom. The material of construction of the envelope is 304 stainless steel.

Figure 10 is a photograph of the readout panel showing the oscilloscope with trace. The recorder is recessed into the table top.

Figure 11 is a record of the residual gases at a pressure about 10^{-8} to 10^{-9} Torr. (The exact pressure is not known since pump current has been used to gauge the pressure.) As shown on the recording, hydrogen is the most prevalent gas. It has been observed that water can be readily detected before bakeout but not after bakeout, indicating that the H_2O has largely been removed.

OPTICAL PYROMETER

To review briefly, the optical pyrometer to be used on this project measures temperature by electronically comparing the radiation received from the creep specimen with that from a calibrated reference lamp.

For some time daily checks have been made with the optical pyrometer and the bench standard. These checks have involved moving of the various units to insure that this does not have an effect on the operation of the instrument. Results indicated that moving the various units (pyrometer, bench standard, etc.) did not affect the temperature readings. However, a small increase in temperature with time (1°F in four days) was detected. This increase is believed to be due to aging of the filament in the standard lamp used for calibration.

Figure 12 shows the equipment operating in conjunction with the bench standard. The optical pyrometer and associated electronics are mounted on the tripod. The standard is enclosed in a cabinet placed on the bench. The cart adjacent to the tripod and bench houses the D.C. power source and associated components required to maintain filament currents to one part in 1,000-10,000.

OPTICAL EXTENSOMETER

The optical pyrometer which is to be used in this program is a double collimator system having one objective movable with respect to the other. The movable objective is attached to a special micrometer slide with a sensitivity of 50 micro-inches. A two-inch gauge length will be used.

The optical extensometer has just been received and bench testing is now underway to develop the necessary operating techniques. While a detailed discussion of this unit will be given at a later date, a photograph of the equipment is shown in Figure 13.

ALLOY SELECTION

The present schedule calls for the generation of creep design-type data for space power systems. The materials being considered for creep testing are listed below:

Sheet Specimens

W (unalloyed)-arc melted

W-25 Re

Sylvania A

Bar Specimens

TZM

TZC

ST-222

Cb-132M

AS-30

In addition to the above alloys, FS-85 will be used in initial check-out tests of equipment and procedures.

Three of the alloys--W, W-25 Re, TZM--are on order. These alloys are being purchased to the detailed material specifications for sheet and forgings assembled in the Appendix. The specifications for the forging material can also be used when ordering cross rolled plate for those turbine alloys which cannot readily be obtained as forgings, i.e., TZC, ST-222, Cb-132M, AS-30.

WORK-IN-PROGRESS

Air, water, and electrical power facilities are now available for the creep laboratory. Until a water softener can be incorporated into the water system, distilled water will be used as the coolant for the first tests.

Specimens of FS-85 sheet are being machined and will be creep tested for comparison with other results run under similar conditions. The alloy procurement program is continuing, as is the bench testing of the residual gas analyzers, optical pyrometer, and optical extensometer.

John C. Sawyer
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E. B. Evans
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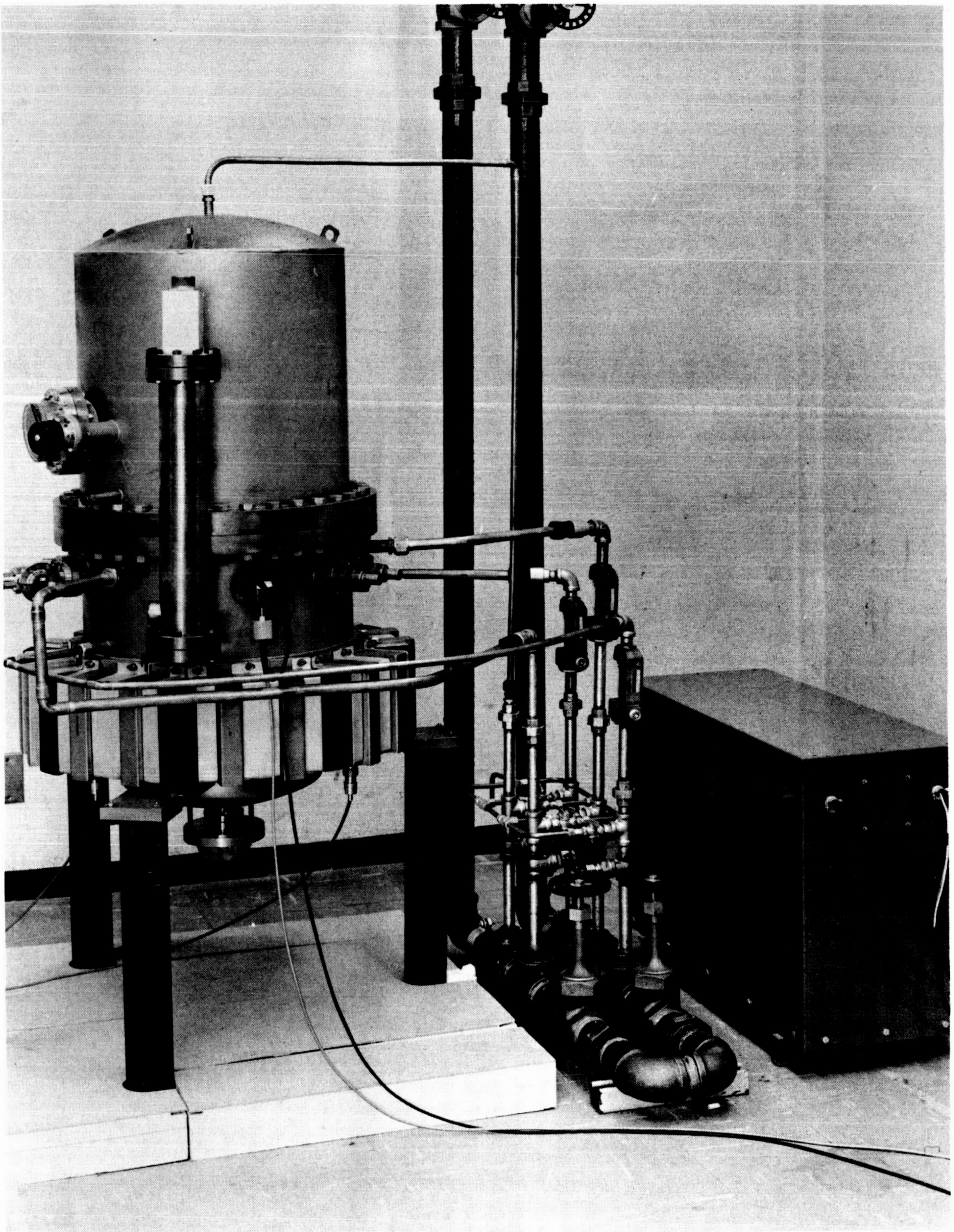

FIGURE 1 VACUUM CREEP UNIT

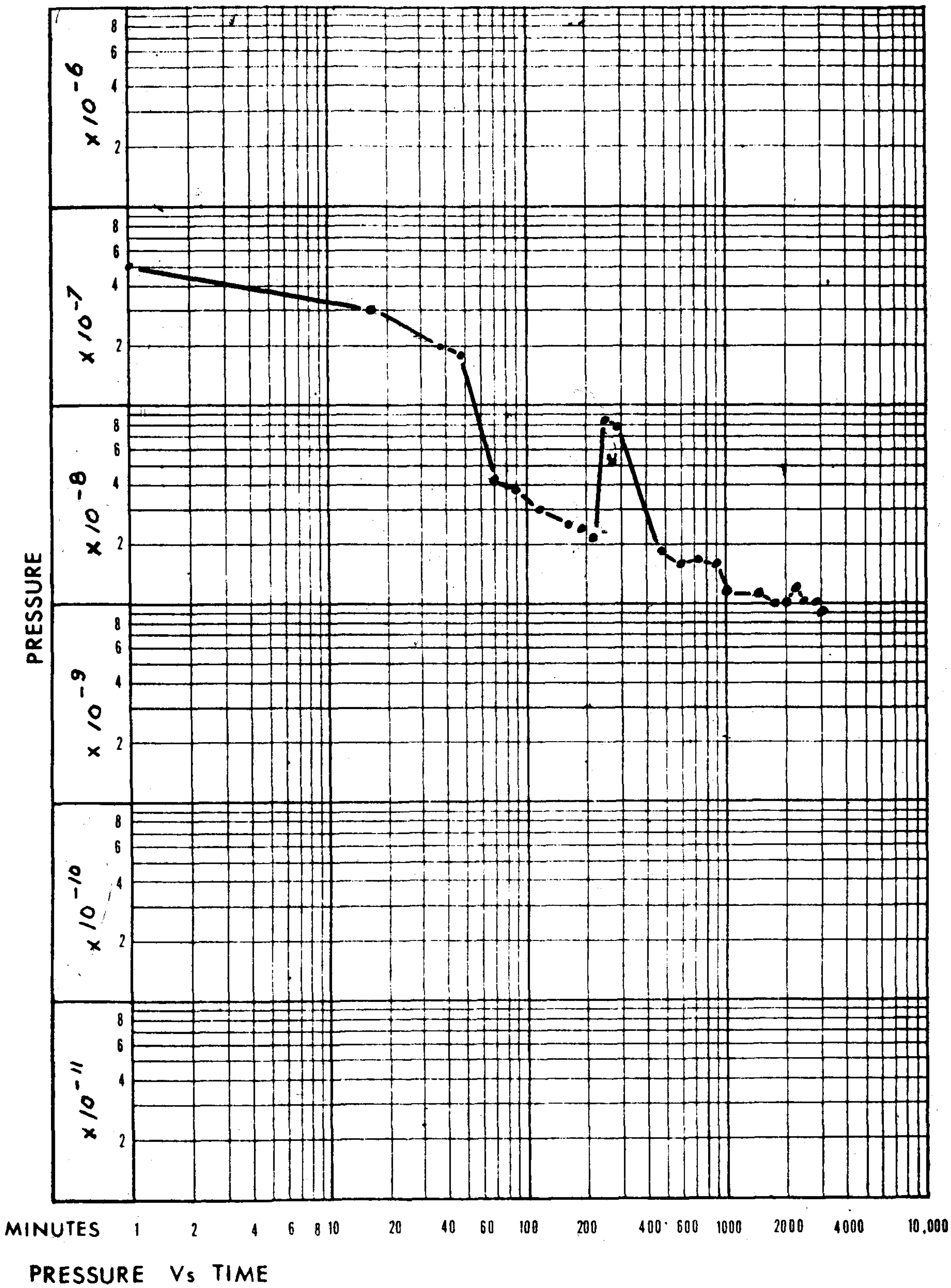

FIGURE 2 PRESSURE VS TIME FOR TEST AT 3500°F

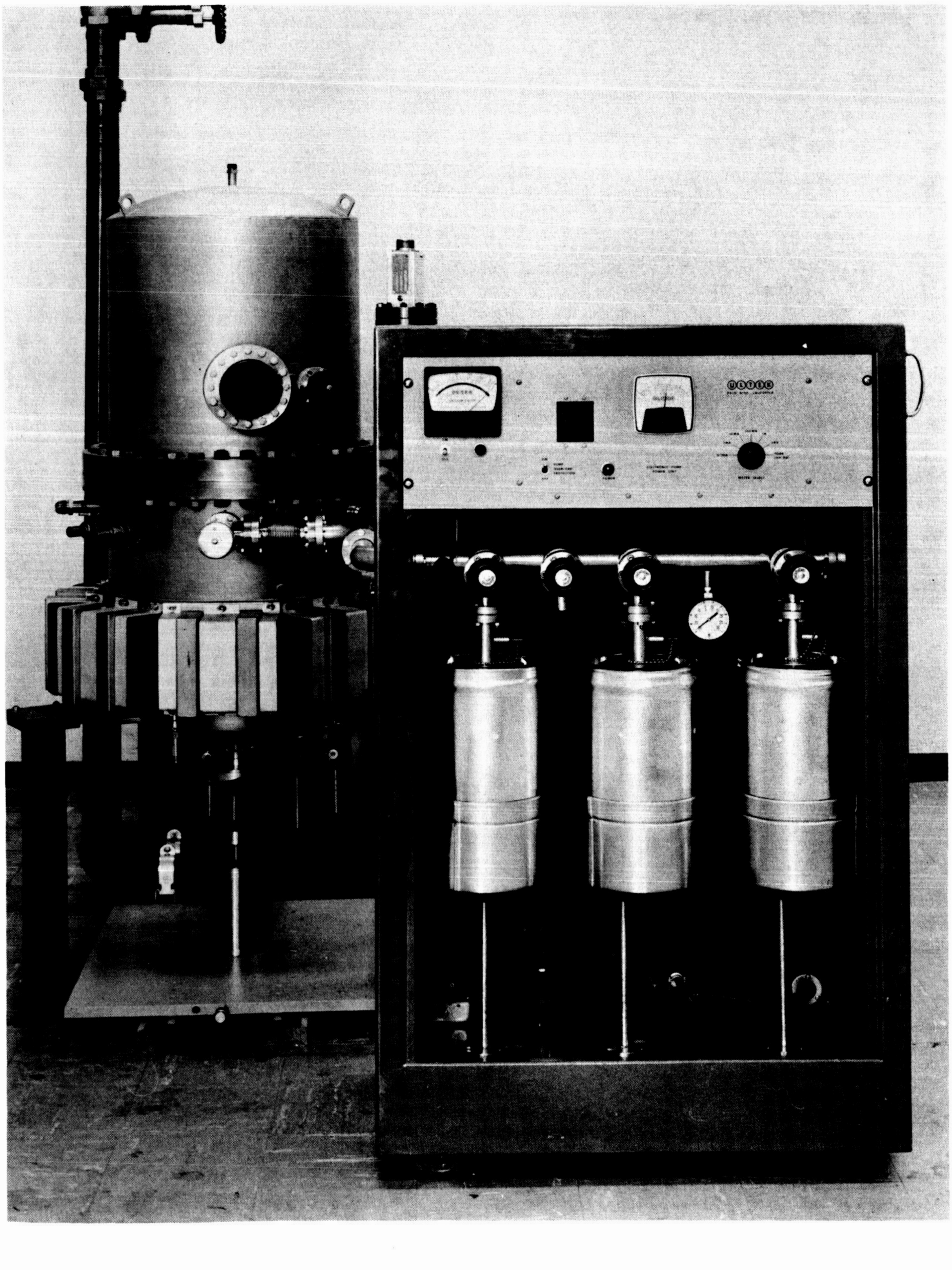

FIGURE 3 ROUGHING CART

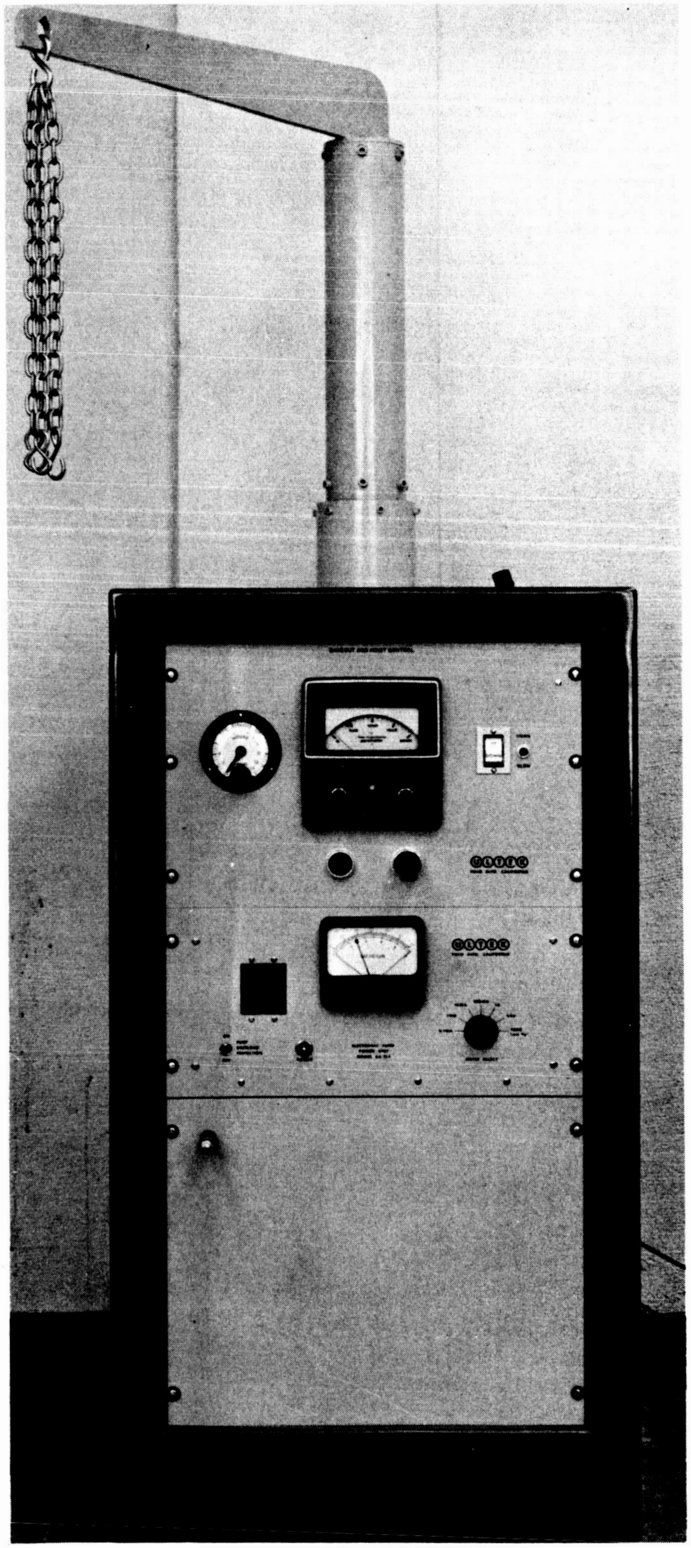

FIGURE 4 HOIST AND BAKEOUT CART

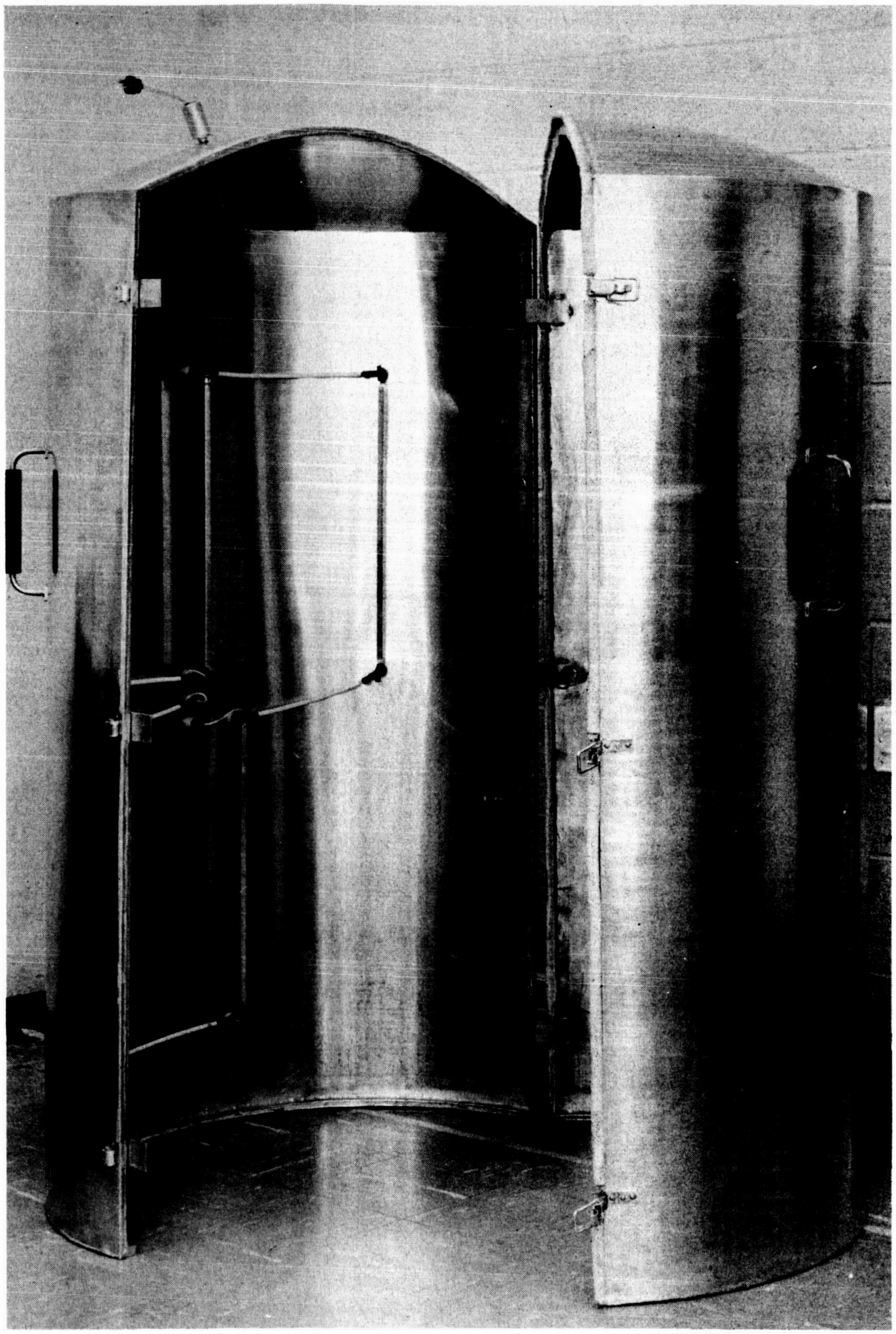

FIGURE 5 BAKEOUT OVEN

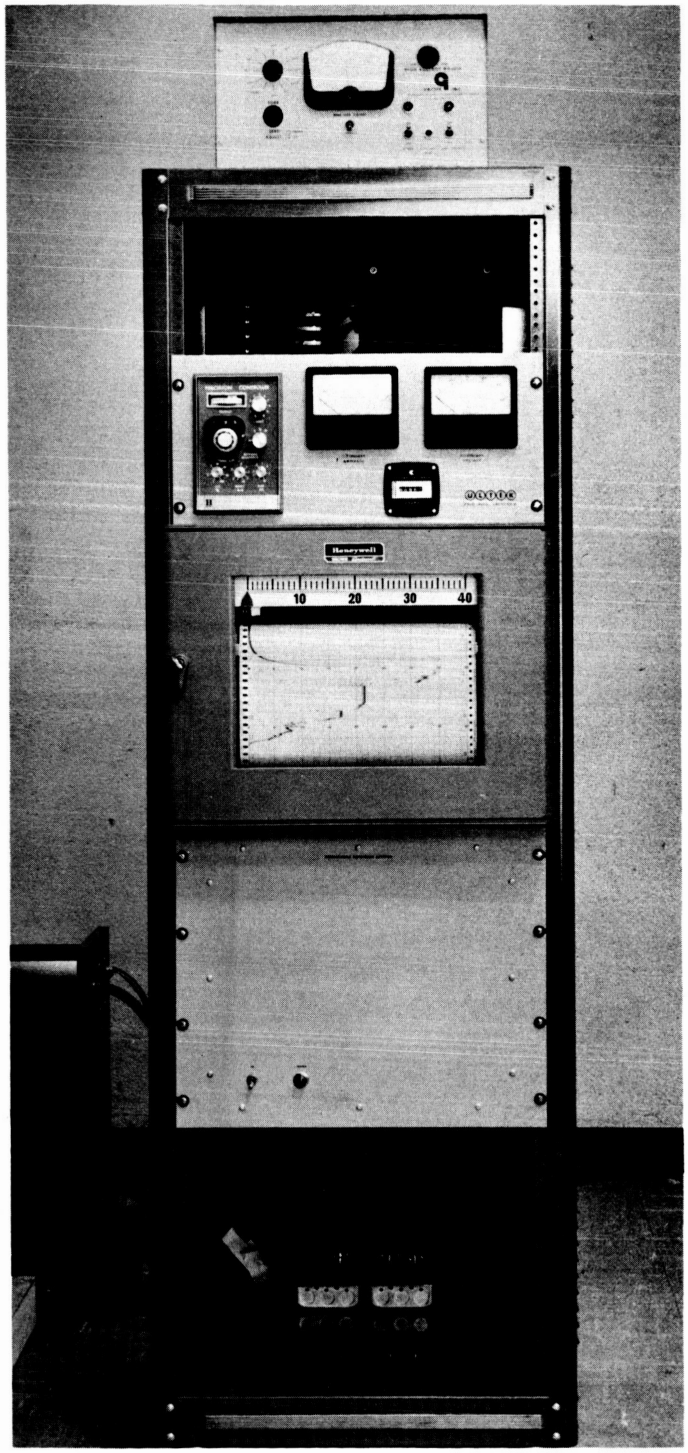

FIGURE 6 TEMPORARY CONSOLE

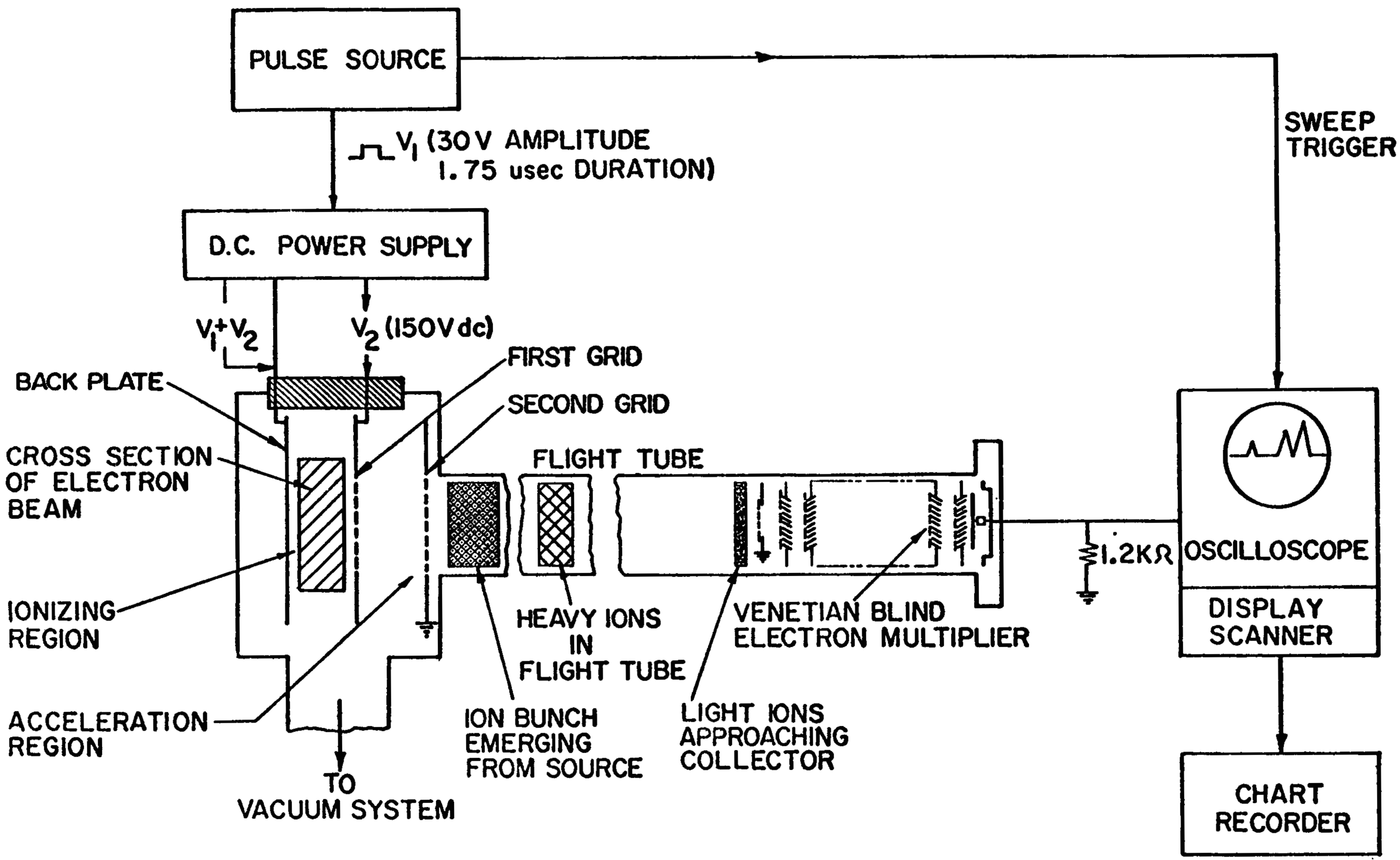

FIGURE 7 SCHEMATIC DIAGRAM OF THE ANALYZER HEAD

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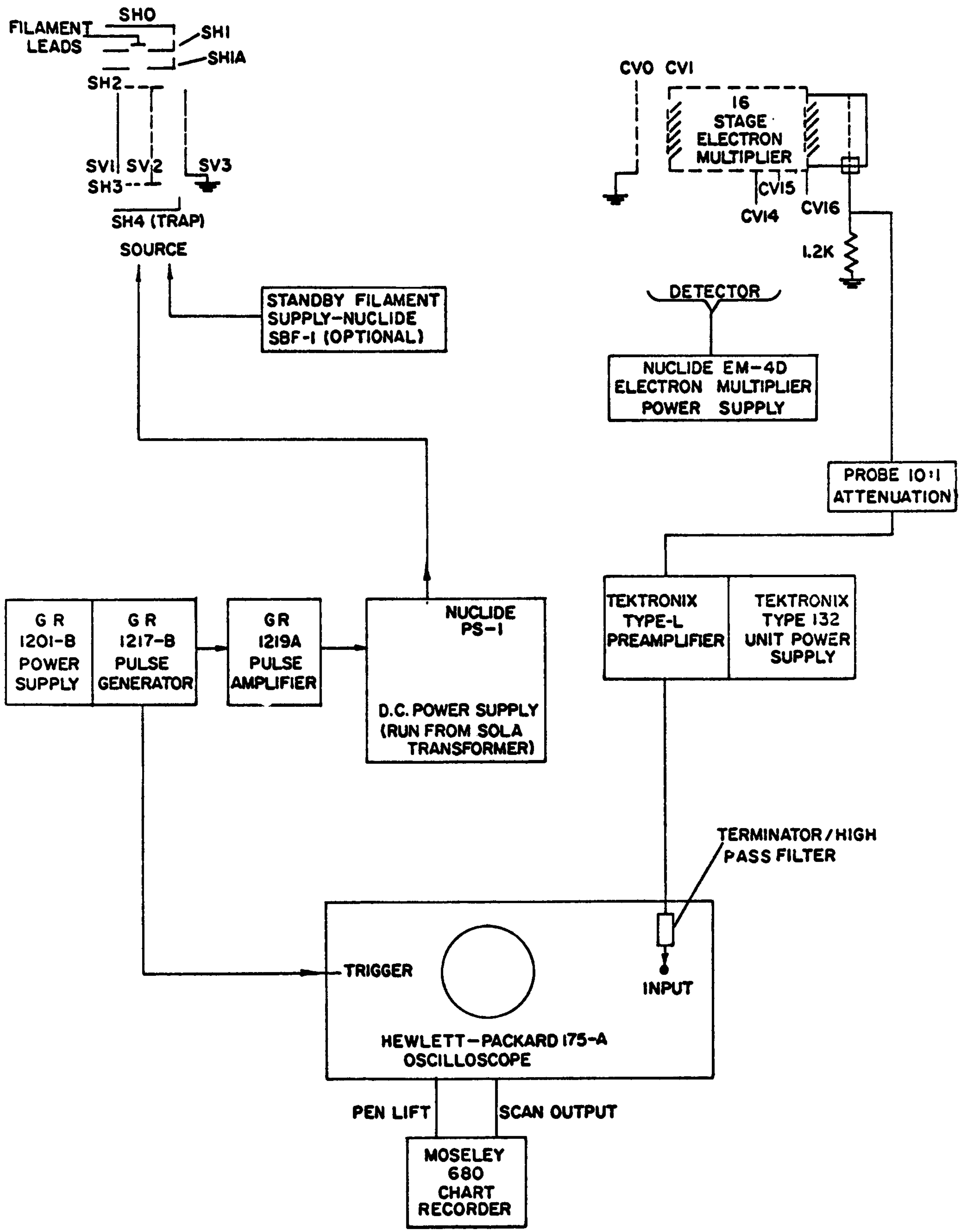

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FIGURE 8 BLOCK DIAGRAM OF RESIDUAL GAS ANALYZER

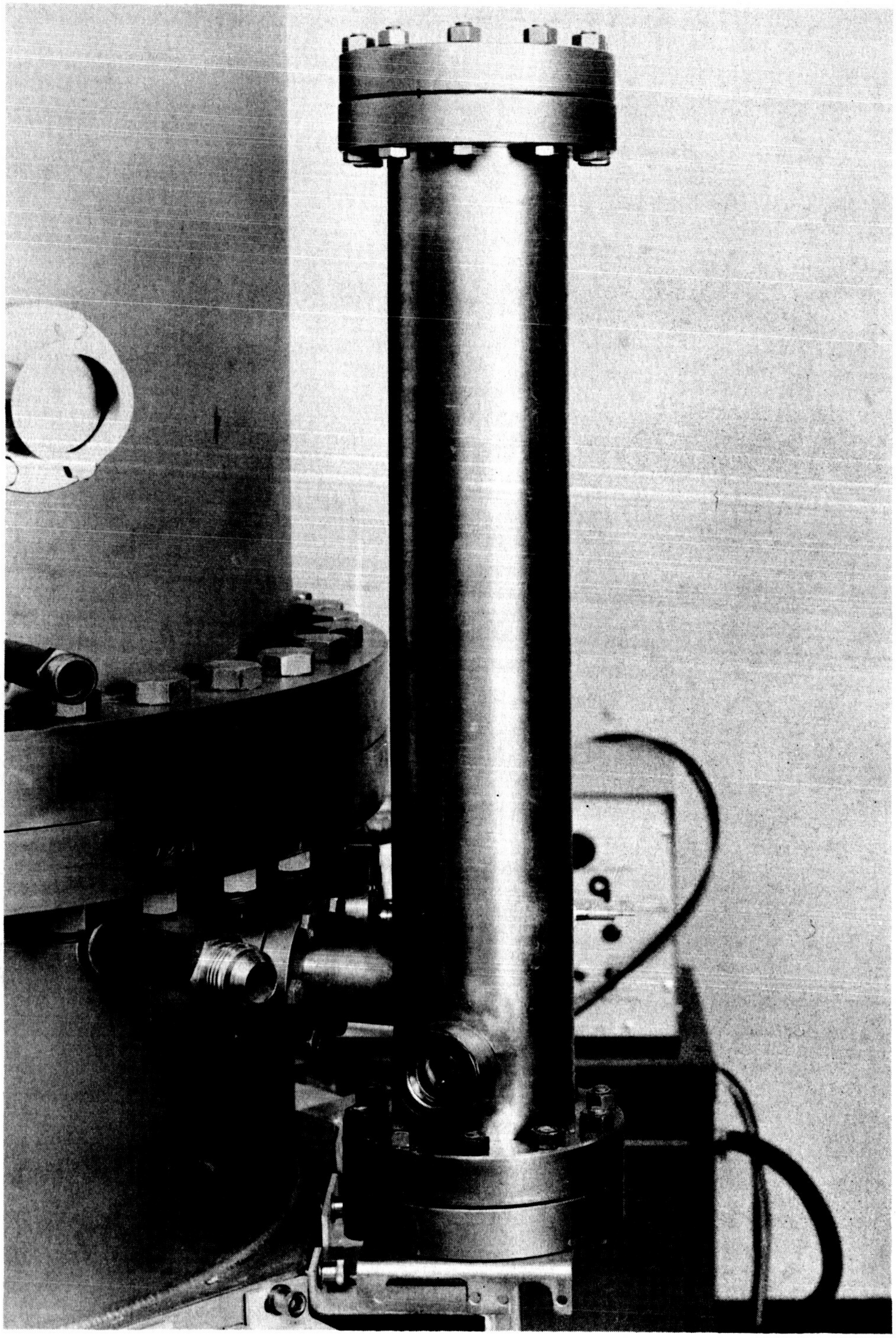


FIGURE 9 FLIGHT TUBE ATTACHED TO CREEP UNIT

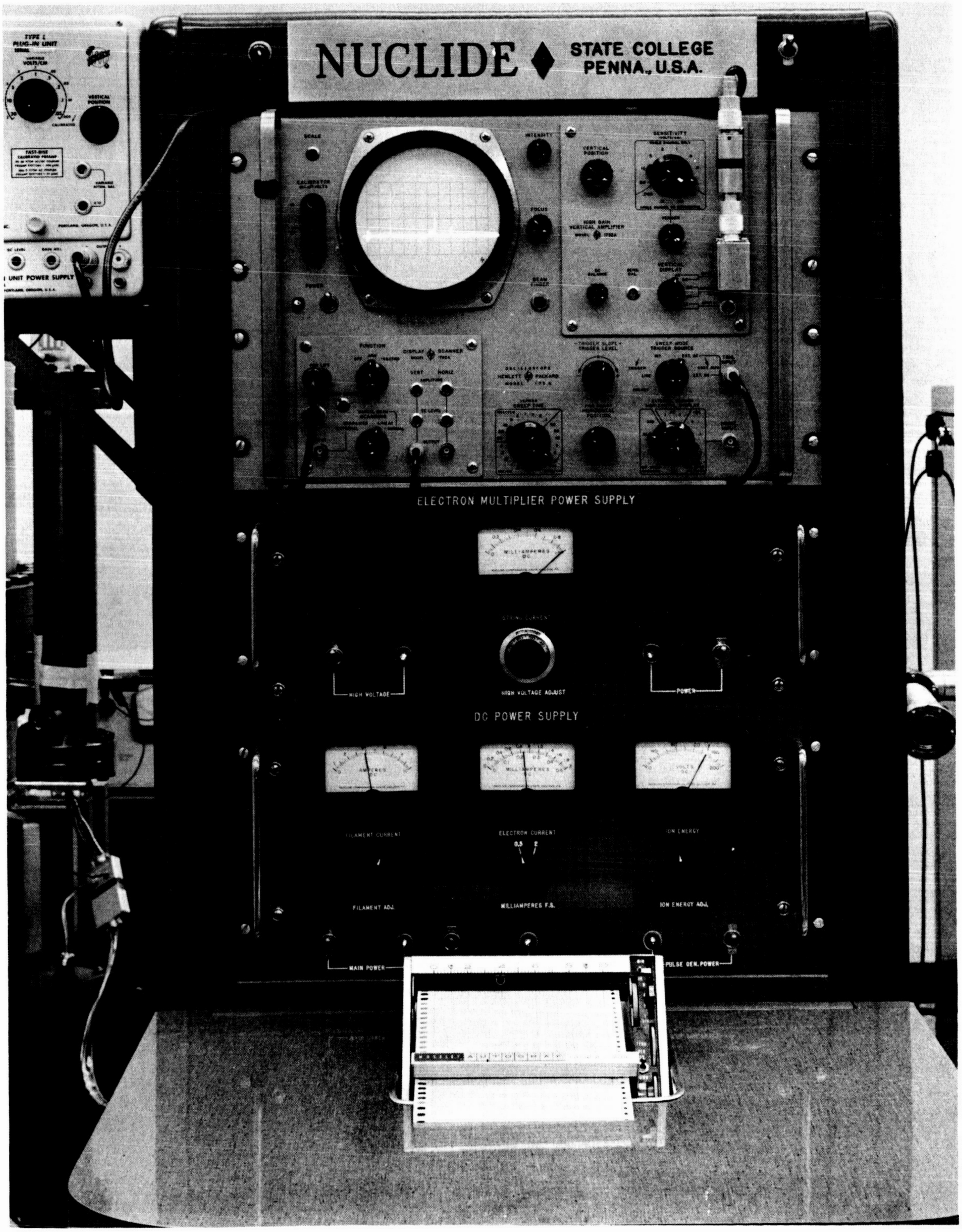


FIGURE 10 READOUT PANEL FOR RESIDUAL GAS ANALYZER

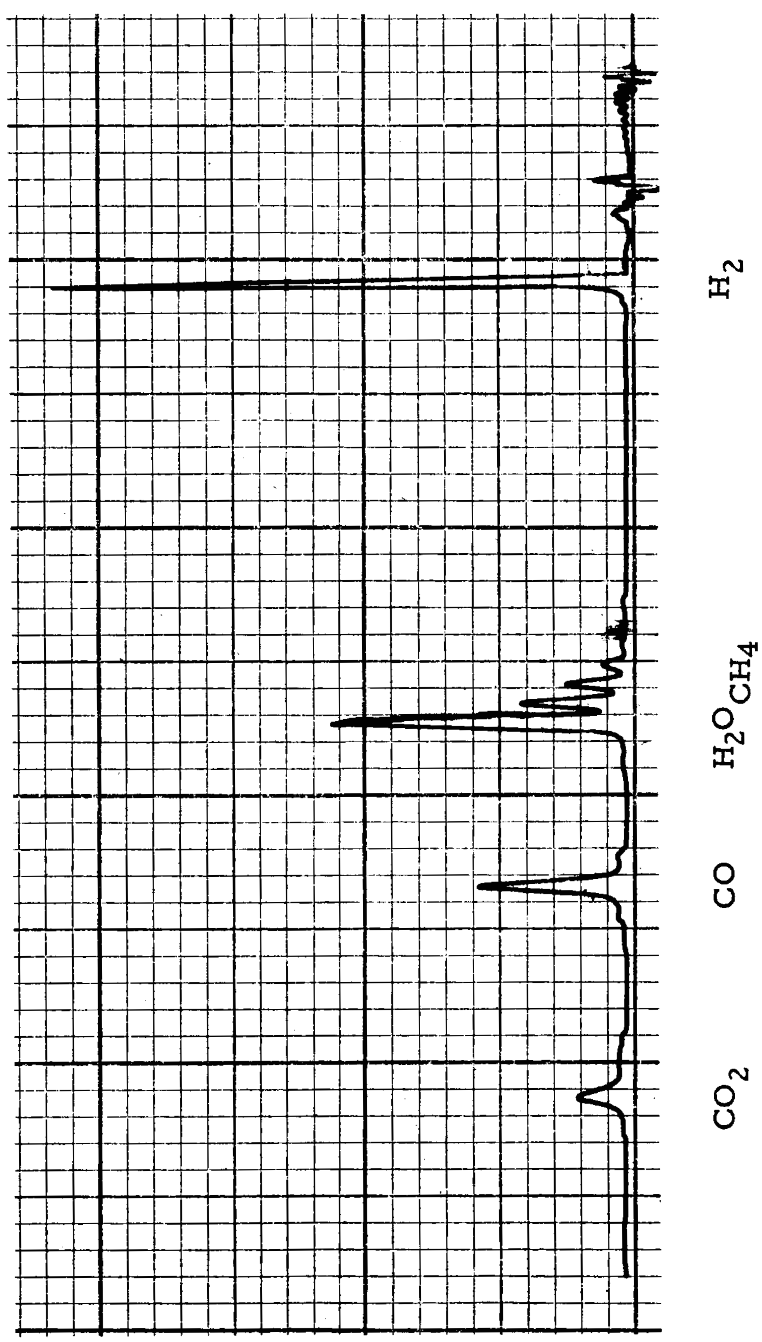


FIGURE 11 TRACE OF 10^{-8} TORR VACUUM WITH RESIDUAL GAS ANALYZER

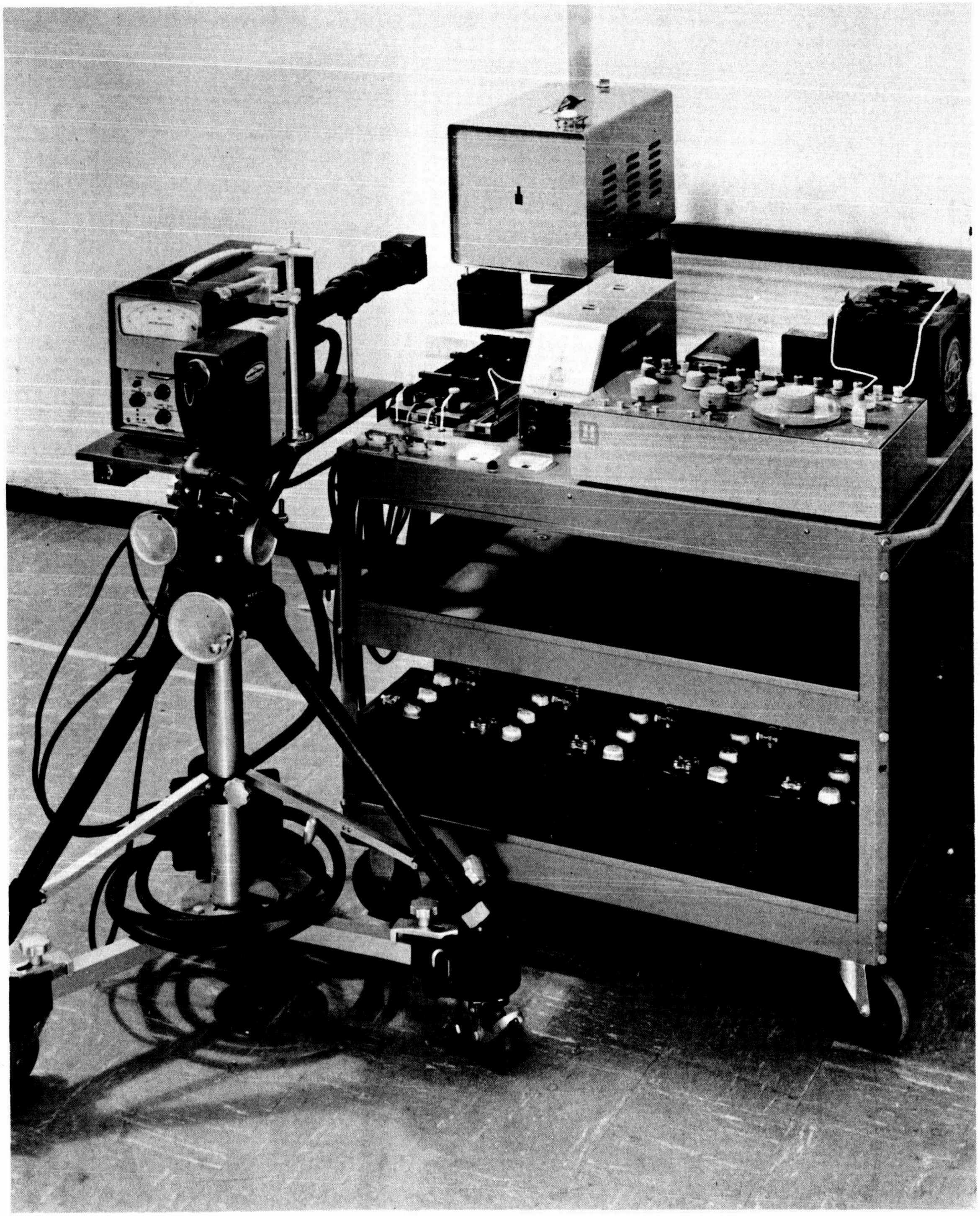


FIGURE 12 OPTICAL PYROMETER WITH BENCH STANDARD

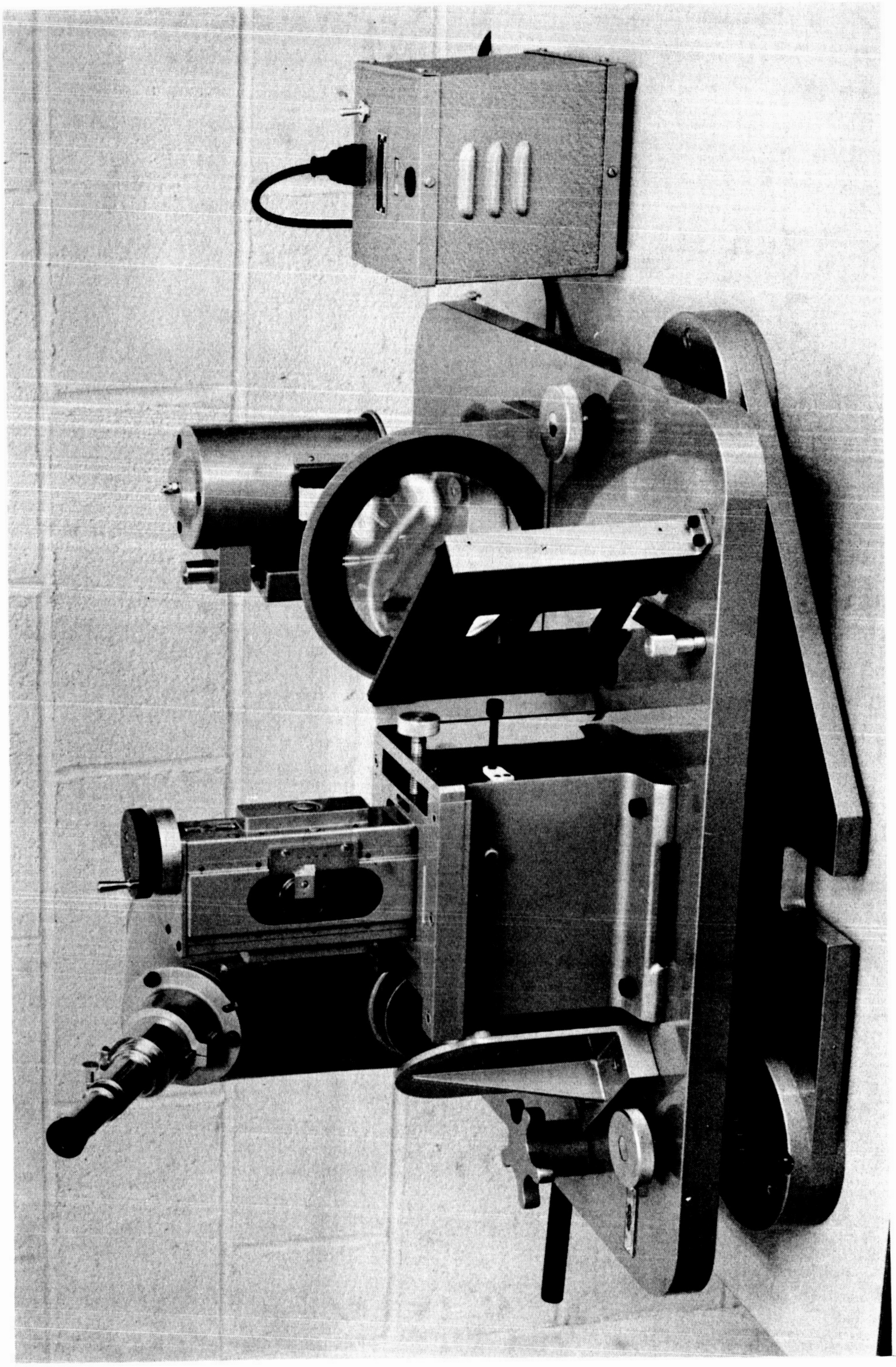


FIGURE 13 OPTICAL EXTENSOMETER

APPENDIX: MATERIAL SPECIFICATIONS

DEVELOPMENT MATERIAL SPECIFICATION

REFRACTORY METAL SHEET MATERIAL - NOMINAL 0.030 INCH THICKNESS

1.0 APPLICATION: TRW is to perform a series of high temperature, high vacuum creep tests on available refractory metal alloys. As part of the testing program, refractory sheet material suitable for tubing components for space power systems will be screened by 1000-hour creep tests. Sheet material with the highest projected strength will then be creep tested for 10,000 hours. Maximum test temperatures will be 3200°F for the tubing alloys. The objective of the tests is to generate valid design data for advanced space power systems.

2.0 MATERIALS:

2.1 DESIGNATION:

Sheet Alloys

FS-85 (Cb-27Ta-12W-0.6Zr)

W-(pure) arc-melted

W-25 Re

Sylvania A (W-base) composition proprietary information.

2.2 FORM: Sheet material 0.030 $^{+0.005}_{-0.000}$ inch thickness. Minimum width 12 inches.

3.0 MANUFACTURE:

3.1 CONSOLIDATION PROCESS: The manufacturer shall furnish, where possible, complete information concerning the processing of the cast ingot into sheet material. Intention of the manufacturer to withhold proprietary information concerning processing details should be clearly stated in his quotation. Processing data includes information such as extrusion temperature, extrusion ratio, forging or rolling temperatures, intermediate annealing temperatures and the final condition of the material (cold-worked, recrystallized, etc.).

3.3 FINAL CONDITION: The final metallurgical structure of the sheet material is left to the judgement of the manufacturer. Our requirements are that the best possible material in its most creep resistant form be used in evaluating each alloy.

3.4 REPORT: The manufacturer will furnish a report (five copies) of the thermal-mechanical history of the material supplied, reporting all pertinent details, except proprietary information.

4.0 CHEMICAL PROPERTIES AND TESTS:

4.1 CHEMICAL COMPOSITION - Alloying elements. Alloy additions for each alloy will fall within the range specified for that particular alloy. Composition ranges, in weight percent, are given below:

Refractory Alloy	Alloying Addition	Composition Range, Weight Percent
FS-85 (columbium-base)	Tantalum	26.0 - 28.0
	Tungsten	11.0 - 13.0
	Zirconium	0.8 - 0.4
W-(pure)	none	
W-25 Re	Rhenium	24.0 - 26.0
Sylvania	proprietary information	

4.2 INTERSTITIAL ELEMENTS: The interstitial levels shall not exceed the following amounts:

Interstitial Element	Maximum Level		Standard Method of Analysis
	Arc-Melted Tungsten	All Alloys	
Oxygen	60	300 ppm	Vacuum gas fusion
Carbon	60	100 ppm	Leco conductometric
Nitrogen	30	100 ppm	Microkjeldahl
Hydrogen	20	20 ppm	Vacuum fusion

4.3 METALLIC IMPURITY LEVEL: The metallic impurity levels of the alloys, as determined spectrographically, shall not exceed the following values:

Impurity Element	Maximum Levels	
	Arc-Melted Tungsten	All Alloys
Al	20 ppm	50 ppm
Co	10 ppm	100 ppm
Cr	10 ppm	50 ppm
Cu	10 ppm	150 ppm
Fe	25 ppm	150 ppm
Mg	10 ppm	50 ppm
Mn	15 ppm	100 ppm
Ni	10 ppm	150 ppm
Pb	20 ppm	50 ppm
Si	25 ppm	150 ppm
Sn	40 ppm	50 ppm
Ti	20 ppm	200 ppm
V	30 ppm	150 ppm
Mo*	100 ppm	500 ppm
Cb*	100 ppm	500 ppm

*Limit unless given as an alloying addition in section 4.1.

4.4 CHECK ANALYSIS: An analysis of each ingot shall be made by the manufacturer to determine the amount of alloying elements (4.1), interstitial elements (4.2), and metallic impurities (4.3) present. The results shall be included in the report on the history of the material (3.4).

5.0 QUALITY:

5.1 SURFACE CONDITION: The finish on the sheet material shall be clean and free of contamination. Cracks, fissures, or imperfections revealed by visual or dye penetrant tests will be cause for rejection of material. Surface roughness should be less than 200 RMS. A matte suface finish due to acid or caustic cleaning is acceptable.

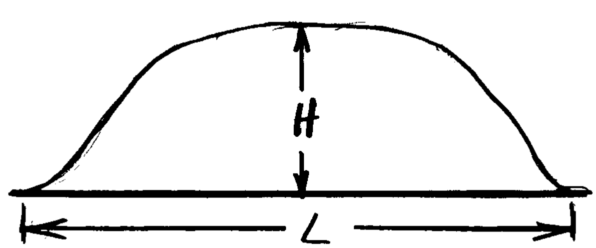
5.2 EDGE CRACKS AND INTERNAL DEFECTS: Edge cracks shall not exceed 1/8 inch in length and shall not occur further than 1/8 inch away from the edge. Internal defects, as revealed by metallography, shall not exceed one percent of the total sheet surface.

5.3 FLATNESS: Deviation from a flat plane shall not exceed five percent when determined by the following criteron.

Percent Flatness = $\frac{H}{L}$ X 100

Where: H is the maximum distance between a flat reference surface and the lower surface of the sheet or plate.

L is the minimum distance between points of contact of the sheet or plate with the flat reference surface.



General bowing of the sheet is acceptable if the bow can be eliminated by slight pressure on the high point of the sheet without causing buckling ripples, or an "oil can" effect.

6.0 PACKING AND MARKING:

6.1 PACKING: The sheet material should be packed in such a manner as to prevent bending or damage during shipment. Each container should be conspicuously marked, including contents, TRW's purchase order number and other necessary information.

6.2 MARKING: Each sheet or plate should be marked with this specification number, the commercial designation, heat number, manufacturer's identification, and the nominal thickness. The marking should be sufficiently stable to withstand ordinary handling.

7.0 ACCEPTANCE OR REJECTION:

7.1 MATERIAL ACCEPTANCE: Acceptance of each lot of material is contingent upon results of chemical analysis and visual super-zyglo fluorescent dye penetrant inspection by TRW after delivery.

7.2 MATERIAL REJECTION: Failure to meet one or more of the requirements set forth in this specification shall be sufficient cause for rejection of said material. Prior written approval by TRW authorizing shipment of any deviated material shall not indicate acceptance.

7.3 DEVIATION FROM SPECIFICATION: Written authorization by TRW is required for acceptance of material that does not meet one or more of the requirements of this specification.

DEVELOPMENT MATERIAL SPECIFICATION

REFRACTORY METAL FORGED DISKS - NOMINAL 10 INCH DIAMETER

1.0 APPLICATION: TRW is to perform a series of high temperature, high vacuum creep tests on available refractory metal alloys. As part of the testing program, forged disks of high strength refractory alloys suitable for high speed turbine wheels will be screened by 1000-hour creep tests. Forged material with the highest projected strength-to-weight ratio will then be creep tested for 10,000 hours. Maximum test temperature for the forged material will be 2200°F. The objective of the tests is to generate valid design data for advanced space power systems.

2.0 MATERIALS:

2.1 DESIGNATION:

Turbine Alloys

TZM - (Mo-0.5Ti-0.08Zr)

TZC - (Mo-1.25Ti-0.15Zr-0.15C)*

ST-222 - (Ta-11.2W-2.8Hf-0.1C)

Cb - 132M - (Cb-15W-20Ta-5Mo-2Zr-.13C)

AS-30 - (Cb-20W-1Zr-0.1C)

*Allowable carbon range on this material 0.03 to 0.15 percent.

2.2 FORM: The material will be forged into a pancake disk forging of 9.8 inches diameter minimum and 0.7 inch thickness minimum.

3.0 MANUFACTURE:

3.1 CONSOLIDATION PROCESS: Vacuum arc melted and/or electron beam melted ingots only. Written authorization by TRW is required for acceptance of powder metallurgy material.

3.2 BREAKDOWN AND FORMING PROCESS: The manufacturer shall furnish, where possible, complete information concerning the processing of the cast ingot into forged material. Intention of the manufacturer to withhold proprietary information concerning processing details should be clearly stated in his quotation. Processing data includes information such as extrusion temperature, extrusion ratio, forging or rolling temperatures, intermediate annealing temperatures and the final condition of the material (cold-worked, recrystallized, etc.).

3.3 FINAL CONDITION: The final metallurgical structure of the forged material is left to the judgement of the manufacturer. Our requirements are that the best possible material in its most creep resistant form be used in evaluating each alloy.

3.4 REPORT: The manufacturer will furnish a report (five copies) of the thermal-mechanical history of the material supplied, reporting all pertinent details, except proprietary information.

4.0 CHEMICAL PROPERTIES AND TESTS:

4.1 CHEMICAL COMPOSITION: Alloying elements. Alloying additions for each alloy will fall within the range specified for that particular alloy. Composition ranges, in weight percent, are given below:

Refractory Alloy	Alloying Addition	Composition Range, Weight Percent
TZM Molybdenum base	Titanium Zirconium Carbon	0.40 - 0.55 0.06 - 0.12 0.01 - 0.04
TZC Molybdenum base	Titanium Zirconium Carbon	1.00 - 1.60 0.15 - 0.30 0.03 - 0.15
ST-222 Tantalum base	Tungsten Hafnium Carbon	11.0 -12.0 2.0 - 3.0 0.010- 0.014
Cb-132M Columbium base	Tungsten Tantalum Molybdenum Zirconium Carbon	13.5 -16.5 18.5 -21.5 4.5 - 5.5 1.75 - 2.25 0.11 - 0.15
AS-30 Columbium base	Tungsten Zirconium Carbon	19 -21 0.75 - 1.25 0.75 - 0.125

4.2 INTERSTITIAL ELEMENTS: The interstitial levels shall not exceed the following amounts:

Interstitial Element	Tantalum Alloys	Molybdenum Alloys	Columbium Alloys	Standard Method of Analysis
Oxygen	50 ppm	50 ppm	250 ppm	Vacuum gas fusion
Nitrogen	50	50	100	Microkjeldahl
Hydrogen	10	10	20	Vacuum fusion

4.3 METALLIC IMPURITY LEVEL: The metallic impurity levels of the alloys as determined spectrographically, shall not exceed the following values:

Molybdenum Alloys	Tantalum Alloys	Columbium Alloys
Al - 80 ppm	Al - 50 ppm	Co - 100 ppm
Co - 25 ppm	Co - 25 ppm	Cu - 100 ppm
Cr - 25 ppm	Cr - 25 ppm	Fe - 100 ppm
Cu - 25 ppm	Cu - 25 ppm	Mn - 100 ppm
Fe - 50 ppm	Fe - 50 ppm	Ni - 200 ppm
Mg - 25 ppm	Mg - 25 ppm	Pb - 50 ppm
Mn - 50 ppm	Mn - 50 ppm	Si - 200 ppm
Ni - 50 ppm	Ni - 50 ppm	Ti - 300 ppm
Pb - 50 ppm	Pb - 50 ppm	V* - 150 ppm
Si - 50 ppm	Si - 50 ppm	Mo* - 200 ppm
	Ti -100 ppm	
V - 50 ppm	V - 50 ppm	
Sn -100 ppm	Sn -100 ppm	
Cb -100 ppm	Mo -100 ppm	
	Cb -100 ppm	

*Limit unless major alloying constituent.

4.4 CHECK ANALYSIS: An analysis of each ingot shall be made by the manufacturer to determine the amount of alloying elements (4.1), interstitial elements (4.2), and metallic impurities (4.3) present. The results shall be included in the report on the history of the material (3.4).

5.0 QUALITY:

5.1 SURFACE CONDITION: The surface of the disk forging may be of a machined, ground, or grit blasted finish. Surface roughness should be less than 200 RMS.

5.2 SURFACE DEFECTS: There should be no surface or edge cracks on the disk forging, as revealed by visual and dye penetrant inspection.

5.3 INTERNAL DEFECTS: Ultrasonic inspection will be used to judge the quality of the forged disk. To be acceptable, the material must meet acceptance limits of class A areas, as defined in NASA specification LRC-4, Ultrasonic Testing of Forgings. Class A areas are defined as having no indications of flaws in excess of a 5/64-inch diameter flat bottomed hole. Multiple indications greater than 3/64-inch diameter flat bottomed hole may not be closer than 1.0 inch. For complete details refer to the NASA specification on ultrasonic inspection.

6.0 PACKING AND MARKING:

6.1 PACKING: The forged disks should be packed in such a manner as to prevent damage during shipment. Each container should be conspicuously marked, including contents, TRW's purchase order number and other necessary information.

6.2 MARKING: Each forged disk should be marked with this specification number, the commercial designation, heat number, and manufacturer's identification. The marking should be sufficiently stable to withstand ordinary handling.

7.0 ACCEPTANCE OR REJECTION:

7.1 MATERIAL ACCEPTANCE: Acceptance of each lot of material is contingent upon results of chemical analysis visual super-zyglo fluorescent dye penetrant inspection and ultrasonic inspection by TRW after delivery.

7.2 MATERIAL REJECTION: Failure to meet one or more of the requirements set forth in this specification shall be sufficient cause for rejection of said material. Prior written approval by TRW authorizing shipment of any deviated material shall not indicate acceptance.

7.3 DEVIATION FROM SPECIFICATION: Written authorization by TRW is required for acceptance of material that does not meet one or more of the requirements of this specification.

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